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2. Patent application number *(The Patent Office will fill in this part)*	0316577.6	15 JUL 2003
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Patents ADP number *(if you know it)*	798181013	
If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM	
4. Title of the invention	ATOMIC FORCE MICROSCOPE	
5. Name of your agent *(if you have one)*	STEVENS HEWLETT & PERKINS	
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Patents ADP number *(if you know it)*	1545003	
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Description	12
Claim *(s)*	4
Abstract	
Drawing *(s)*	3 + 3

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Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature C. Williams Date 15/07/2003

Agents for the Applicant

12. Name and daytime telephone number of person to contact in the United Kingdom — Dr CEILI WILLIAMS; 020 7404 1955

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ATOMIC FORCE MICROSCOPE

This invention relates to the field of atomic force microscopes, to cantilever probes therefor and to a method of operating such microscopes. In particular, it relates to an atomic force microscope that is operable in constant height mode.

The atomic force microscope (AFM), or scanning force microscope (SFM), was invented in 1986 by Binnig, Quate and Gerber. Like all other scanning probe microscopes, the AFM is based on the principle of mechanically scanning a nanometric probe over a sample surface in order to acquire an "interaction map" of the sample. The interaction force in this case is simply the molecular interaction between the sample and the tip of a sharp probe attached to a cantilever spring. When the probe tip is brought into close proximity with the sample, the cantilever bends in response to the molecular interaction force. Images are collected by scanning the sample relative to the probe and measuring the deflection of the cantilever as a function of lateral position. An optical lever technique is usually used to measure this bending. Since the cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be deduced.

The AFM is usually operated in one of two modes. In constant force mode, feedback enables a positioning piezoelectric driver to move the sample (or probe) up or down in response to any change in the interaction force that is detected. In this way, the interaction force may be held relatively steady and a fairly faithful topographical image of the sample is obtained. Alternatively the AFM may be operated in constant height mode. No, or very little, adjustment of the vertical height of the sample or probe is imparted during the scan. In this context, adjustment of the vertical height means that a translation is applied either to an actuator connected to the

cantilevered probe or to the sample itself. There remains therefore a degree of freedom for the probe tip to move up and down as the degree of cantilever bend is varied. In constant height mode, topographical changes to the sample are indistinguishable from interaction force variations in that either or both will cause the cantilever spring to bend.

In addition to these differing feedback regimes, image contrast is usually obtained in one of three different ways. In contact mode the tip and sample remain in close contact, i.e. in the repulsive regime of the molecular interaction, as scanning proceeds. In tapping mode an actuator drives the cantilever in a "tapping" motion at its resonant frequency. The probe tip therefore only contacts the surface for a very small fraction of its oscillation (tapping) period. This dramatically shortened contact time means that lateral forces on the sample are very much reduced and the probe is therefore less destructive to the specimen as the scan is taken. It is consequently much used for imaging sensitive biological specimens. Oscillation amplitude is generally held constant using a feedback mechanism. In non-contact operation the cantilever is oscillated above the sample at such a distance that the molecular interaction force is no longer repulsive. This mode of operation is however very difficult to implement in practice.

Recent advances in probe microscopy have led to much faster data collection times. With faster scan techniques, such as that described in PCT patent application publication number WO 02/063368, finite probe responsivity is increasingly becoming a limiting factor in image collection times. The probe will not respond instantaneously to a change in sample characteristics and so there is an inherent time delay between, for example, the probe encountering a region of the sample surface with increased height and the system reacting to it. This disadvantage applies to both constant force and constant height modes of AFM operation. It is less severe in constant height mode, which is therefore the preferred mode

of operation for fast scanning techniques, but it is still sufficient to limit unduly the scan speed of the current generation of fast scanning probe microscopes.

In constant force AFM mode, an electronic feedback mechanism is usually employed in order to keep the average interaction force constant. As the scan progresses if there is a change in interaction force (for example caused by a change in sample height) this is first observed by the detection electronics, a feedback signal is then generated, the probe or sample height adjusted in response to this signal and then there is also a finite time taken for the probe (or sample) to settle at its new position. This sequence imposes a limitation on the ultimate speed with which a full image scan can be collected.

The problem is not so restrictive if operating in constant height mode, in which electronic feedback is not normally used to the extent that it is used in constant force AFM. For the interaction force to be measured accurately however the probe tip should, as far as possible, track the contours of the sample surface. This is ensured by exploiting the reaction force developed as the cantilever is bent by the sample surface. That is, as a high region of the sample surface is scanned, the cantilever is increasingly bent upwards and the energy stored in the spring is increased. As the height falls away, a restoring force pushes the cantilever back towards its equilibrium (straight) position, thus maintaining contact with the surface. If however the scan speed is sufficiently fast, the probe will not track the surface but will effectively be thrown upwards over any protuberance from the surface and may start to resonate, or "ring". This in turn gives rise to oscillations in the imaged interaction force.

WO 02/063368, referred to above, describes a scanning probe microscope in which the probe is oscillated at resonance whilst translated in order to interrogate the sample very rapidly with an arrangement of scan lines. The

typical time spacing between pixels is therefore shorter than $1/f_r$, where f_r is the resonant frequency of the probe. On the other hand the time taken (τ_{res}) to respond to a change in topography of the sample surface is based on the effective mass of the probe and the spring constant of the cantilever. If $\tau_{res} > 1/f_r$, then clearly the interaction force will not be measured accurately from scan line to scan line, and certainly not for all image pixels.

There is a perceived need to provide for improved probe responsivity to sample topographic fluctuations or to variations in the interaction force and so to permit AFM microscopy to be performed at faster scanning speeds before image artefacts such as those caused by probe ringing start to degrade image quality.

The present invention provides an atomic force microscope for imaging a sample in accordance with a molecular interaction force between the sample and a cantilever probe, the microscope comprising

driving means arranged to provide relative scanning motion between the probe and the sample surface and capable of bringing the sample and probe into close proximity, sufficient for a detectable interaction to be established between them; and

a probe detection mechanism arranged to measure deflection of the cantilever probe;

characterised in that, the microscope includes additional force (F_{direct}) generating means arranged such that, in operation, an additional force (F_{direct}) is applied to either the sample or the probe, the force (F_{direct}) being directed so as to attract the probe towards the sample or *vice versa*.

In an alternative aspect the present invention provides a probe for use in an atomic force microscope or for nanolithography, the probe comprising a

cantilever and sharpened tip characterised in that the probe also includes a magnetic element located on the cantilever in the region of the tip.

In a third aspect the present invention provides a method of collecting image data from a scan area of a sample with nanometric features wherein the method comprises the steps of:-

(a) Moving a cantilever probe with tip of sub-nanometric dimensions into close proximity with a sample in order to allow an interaction force to be established between probe and sample;

(b) Applying a direct non-contact force (F_{direct}) to either the sample or the probe such that the probe is encouraged to move towards the sample or *vice versa*;

(c) Scanning either the probe across the surface of the sample or the sample beneath the probe whilst providing a relative motion between the probe and surface such that an arrangement of scan lines covers the scan area;

(d) Measuring deflection of the cantilever probe; and

(e) Processing measurements taken at step (d) in order to extract information relating to the nanometric structure of the sample.

Embodiments of the invention will now be described by way of example only and with reference to the accompanying drawings.

Figure 1 is a diagrammatic illustration of the forces involved as a cantilevered probe makes contact with a sample surface in a prior art atomic force microscope.

Figure 2 shows a schematic implementation of an atomic force microscope

in accordance with this invention.

Figure 3 is a diagrammatic illustration of the forces involved as a cantilevered probe makes contact with a sample surface in the AFM of Figure 2.

With reference to Figure 1, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM). The probe comprises a substrate 2 from which a cantilever 3 extends, the cantilever 3 having a sharp probing tip 4 mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force $F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the cantilever 3 is bent upwards from its rest position 5 as the sample is scanned.

The cantilever 3 obeys Hooke's Law for small displacements. Accordingly if, when pressing on the sample, the degree of bending is such as to move the tip 4 a perpendicular distance x from its rest position and the cantilever spring constant is k then the restoring force exerted by the cantilever is kx. The downward force exerted by the tip 4, holding it in position tracking the surface, is thus proportional to kx.

Clearly the responsivity of the probe tip 4 and hence the resolution of the AFM technique depends on the degree of force kx exerted by the cantilever 3 on the sample 1. The greater the force between probe and surface, the greater the responsivity to surface variations. This indicates that a high spring constant k is desirable, particularly if the scan is to be fast. On the other hand, the greater the force, the more likely the probe is to damage the sample. Accordingly prior art AFM cantilever probes must make a fundamental compromise between probe responsivity and the likelihood of

damaging the sample.

Figure 2 shows a schematic implementation of an AFM, indicated generally by 10, that utilises a cantilever probe constructed in accordance with an aspect of this invention. The AFM apparatus 10 shown comprises a plate 12 adapted to receive a sample 14, and which is mounted on one prong of a tuning fork 16. The tuning fork 16 is connected to a piezoelectric transducer 18 and a coarse driving means 20. The piezoelectric transducer 18 is used to drive the sample 14 (together with the plate 12 and fork 16) in three dimensions: *x*, *y* and *z* directions. As is conventional in the field, the *z* axis of a Cartesian coordinate system will be taken to be that perpendicular to a plane occupied by the sample 14. That is, the interaction force is dependent both on the *xy* position of a probe 22 over the sample 14 (the pixel it is imaging), and also of its height above it. A tuning fork control (not shown) is arranged to apply a sinusoidal voltage to the tuning fork 16 and so to excite a resonant or near-resonant vibration within the *xy* plane. The probe 22 is a low-mass AFM cantilever probe and, during a scan, an interaction force is developed between the probe tip and the sample surface. Unlike prior art cantilever probes however, the probe 22 according to this invention has a magnetic bead 24 mounted above the tip. The cantilever component is coated on both sides by a polymeric film and is shaped so as to have a low spring constant, less than 1 Nm^{-1}. A magnet 26 is incorporated within the AFM to provide a magnetic field of sufficient strength to exert a force on the magnetic bead 24. A probe detection mechanism 28 is arranged to measure the displacement of the probe tip and thus the bending of the cantilever 22, which is indicative of molecular interaction force strength. Data collected by the probe detection mechanism 28 is analysed and output to a display 30.

In taking images using the apparatus 10, the sample 14 is first brought into contact with the cantilever probe 22 using the coarse driving means 20. Fine height and initial start position adjustments are made with the piezo

driver 18 whilst the probe detection mechanism 28 measures the cantilever's bending as a result of the probe 22 – sample 14 interaction force. Once the measured bending reaches a desired level, the magnet 26 is switched on and a magnetic field *B* is generated in the vicinity of the probe tip. The magnetic bead 24 interacts with this field, which is directed such that the resultant magnetic force attracts the magnetic bead 24 downwards into the sample 14. The probe tip is therefore held in contact with the sample 14 by the direct action of this magnetic force. With the magnetic field on, the sample surface is scanned beneath the probe 22. In scanning the sample 14 under the probe 22, the tuning fork 16 is set to vibrate into and out of the plane of the Figure (*y* axis). This oscillates the stage on which the sample is mounted. At the same time, the piezo 18 translates the sample 14 in a perpendicular (*x*) direction. Sample oscillation is with a relatively large amplitude, of the order of a few microns. During the course of a scan, readings are continually taken by the probe detection mechanism 28, which, as is standard in the art, is based on an optical lever technique: cantilever bend is measured using laser light reflected from the probe. The output signal from the probe detection mechanism 28 is fed directly to a processor and display 30.

In order to appreciate the features that are necessary to this invention it is helpful to look at a diagrammatic representation of the forces involved while a scan is being performed. This is illustrated in Figure 3, which shows the same set up as Figure 1 and so like components are similarly referenced. With reference to Figure 3, there is shown a sample 1 that is being scanned by a probe of an atomic force microscope (AFM) in accordance with the present invention. The probe comprises a substrate 2 from which a cantilever 3 extends, the cantilever 3 having a sharp probing tip 4 mounted at an end remote from the substrate 2. In preparation for a scan, a downwards force ($F_{external}$) is applied to the probe at its substrate end 2 via its mounting to the AFM, moving the probe tip 4 into contact with the sample 1. In order to maintain contact for the duration of a scan, the force

$F_{external}$ is greater than that required simply to bring the tip 4 into contact with the sample 1. As a result the cantilever 3 is bent upwards from its rest position 5 as the sample is scanned. As before, a force proportional to kx is generated as a result of the cantilever bending and directs the probe tip 4 downwards towards the sample surface.

In addition to the cantilever force kx generated by prior art AFMs, a microscope in accordance with the present invention is also arranged to provide a second attractive force F_{direct} between probe and sample. The total restoring force holding the probe to the surface is now:

$$F_{direct} + \alpha kx,$$

where α is a constant of proportionality for the downwards force arising from the cantilever bend. Ideally, the additional force F_{direct} is much greater than the cantilever bending force kx. Its magnitude should moreover be sufficiently large to bring the probe into contact with the surface, should it lose contact, within approximately one pixel. In the embodiment depicted in Figure 2, the additional force F_{direct} is a magnetic force, provided by applying a magnetic field to a cantilever tip that incorporates a magnetic bead. Clearly therefore the positioning of the magnet within the AFM is not critical, it merely has to be arranged such that there is a downward force component pulling the probe tip 4 into the sample 1.

In order to assist in achieving $F_{direct} >> kx$, the cantilever should have a relatively low spring constant. Typically this should be less than 1 Nm^{-1}, which can be achieved by using a suitably shaped cantilever. This allows the contribution made by the cantilever restoring force αkx to the total force applied to the probe to be neglected. In the present invention, the cantilever force kx is useful only to define the position in space at which the probe sits, i.e. the interaction force between probe and sample, and so enable an image to be collected.

The use of a direct restoring force F_{direct} as opposed to relying on the cantilever force represents a significant improvement over the prior art. By effectively masking the cantilever contribution, the direct force enables the restoring force to have a magnitude that is essentially independent of the position of the cantilever. By way of contrast, the magnitude of the prior art restoring force kx depends on the displacement x of the cantilever from its rest position. Thus high restoring forces are generated at particularly high regions of the sample. This may restore the probe to its surface position quickly, but such rapid movement may also damage the sample. It is very difficult to ensure consistently that samples are not damaged if the restoring force is permitted to vary in this manner. A restoring force implemented in accordance with this invention has a magnitude that is independent of sample height.

It is not essential that the force F_{direct} is a magnetic force, although it should be a force that first does not depend on sample height and secondly can be arranged to be larger than the cantilever restoring force kx. As well as the magnetic bead / magnet arrangement of Figure 2, an electrostatically charged region can, for example, be incorporated in the probe and attracted towards the sample by an electric field.

The cantilever 3 is ideally further adapted to be used in the present invention. Most commercially available AFM cantilevers (for example Si single crystal) have a high quality (Q) factor. If the cantilever has a high Q, it will take a long time to respond to changes and it will ring at its resonant frequency if given a stimulus, such as provided by scanning across a high feature on the sample surface. The present cantilever is therefore selected to have a low Q, ideally such that any induced oscillation is critically damped. This can be done either by judicious selection of its shape or by coating one or both sides with an energy absorbing material, such as a polymer film. The use of a low quality factor means that little energy can be stored in the cantilever spring and so it will not "ring" for long if shocked,

such as when scanning over a high region of the sample surface. Alternatively a low Q factor can be provided if the cantilever is simply immersed in liquid during the scan. In this situation Q ~ 1.

The cantilever, probe tip and any additional component such as the magnetic bead are ideally of low mass. This naturally increases the acceleration of the tip back to the surface for a given restoring force and so better enables the probe to track the surface.

It is to be noted that the apparatus shown in Figure 2 is merely illustrative of an exemplary AFM. There are numerous different embodiments of AFM with which this invention may be implemented. For example, the piezos 18, 20 may be arranged to translate the probe 22 rather than the sample 14, all that is required is that a relative translation is imparted by the scan. Nor is mounting on a tuning fork necessary. This arrangement is simply used in this embodiment in order to illustrate the applicability of this invention to fast scanning techniques that make use of a resonant oscillation. It is equally applicable to slower scanning methods. The probe 22 may alternatively be oscillated in place of the sample 14, although this may cause problems when measuring probe deflection using the optical lever technique.

If a tuning fork 16 is used then it may be one of a number of commercially available forks, or of bespoke design to provide a desired frequency of oscillation. A suitable example is a quartz crystal fork with resonant frequency of 32 kHz. A tuning fork is well suited to this application as it is designed with highly anisotropic mechanical properties. Its resonances are therefore independent and can be individually excited and so limited to only that (or those) in the plane of the sample. Importantly, the fork 16 can be resonated in one direction and scanned in another, without coupling occurring between modes. It therefore permits stable fast motion of the sample 14 as it is interrogated by the probe 22.

The invention is not limited to pure AFM operation, although it is required that there is a force interaction between the probe and the sample surface. This mode of operation can however be combined with microscope components designed to monitor other interactions or interaction indicators between probe and sample. These other interactions may be, for example, optical, capacitative, magnetic, shear force or thermal. Other indicators include oscillation amplitude, either tapping or shear force, capacitance or induced electric currents. These various modes of operation of general probe microscopes are described, for example, in UK patent application number 0310344.7.

The interaction of the probe with the sample surface that is exploited in AFM also makes it possible to affect the properties of the surface and so deliberately "write" information to the sample. This technique is known as nanolithography, and AFMs are widely used for this purpose. For example, by application of a voltage to a conductive cantilever a region of a metallic layer of a sample wafer can be oxidised. Another example exploiting two-photon absorption and polymerisation of a photoresist is described in "Near-field two-photon nanolithography using an apertureless optical probe" by Xiaobo Yin *et al.* in Appl. Phys. Lett. 81(19) 3663 (2002). In both examples the very small size of the probe enables information to be written to an extremely high density. The AFM and cantilever probe of this invention can also be adapted for use in nanolithography. The ability to improve surface tracking with this invention not only offers the potential for faster writing times than previously achieved, but also offers the potential for increased image resolution i.e. write density. To make it more adapted for use in nanolithography the probe tip may be electrically conductive, it may be metal coated in order to increase its optical interaction with the surface or it may be coated with selected molecular species for use in dip pen lithography applications.

CLAIMS

1. An atomic force microscope (10) for imaging a sample (14) in accordance with a molecular interaction force between the sample (14) and a cantilever probe (22), the microscope (10) comprising

   driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity, sufficient for a detectable interaction to be established between them; and

   a probe detection mechanism (28) arranged to measure deflection of the cantilever probe (22);

   characterised in that, the microscope (10) includes additional force (F_{direct}) generating means (24, 26) arranged such that, in operation, an additional force (F_{direct}) is applied to either the sample (14) or the probe (22), the force (F_{direct}) being directed so as to attract the probe (22) towards the sample (14) or *vice versa*.

2. A microscope according to claim 1 characterised in that the additional force (F_{direct}) has magnitude that is independent of the degree of deflection of the cantilever probe (22).

3. A microscope according to claim 2 characterised in that the cantilever probe (22) has spring constant k and the probe (22) properties and additional force (F_{direct}) are arranged such that the additional force (F_{direct}) is much greater than the restoring force kx provided by any cantilever deflection x as it scans the surface of the sample (14).

4. A microscope according to claim 3 characterised in that the probe (22)

has spring constant k that is less than 1 Nm^{-1}.

5. A microscope according to any preceding claim characterised in that the probe (22) comprises a cantilever (3) with low quality factor.

6. A microscope according to claim 5 characterised in that the probe (22) and sample (14) are immersed in liquid during operation of the microscope.

7. A microscope according to claim 5 characterised in that the cantilever (3) is coated on at least one side with a polymeric material.

8. A microscope according to any preceding claim characterised in that the additional force (F_{direct}) generating means (24, 26) comprises a magnet (26) and a magnetic element (24) incorporated in the probe (22).

9. A probe (22) for use in an atomic force microscope or for nanolithography, the probe comprising a cantilever (3) and sharpened tip (4) characterised in that the probe also includes a magnetic element (24) located on the cantilever (3) in the region of the tip (4).

10. A probe according to claim 9 characterised in that the cantilever (3) has a low quality factor.

11. A probe according to claim 10 characterised in that the cantilever (3) is coated on at least one side by a polymeric film.

12. A method of collecting image data from a scan area of a sample (14) with nanometric features wherein the method comprises the steps of:-

    (a) Moving a cantilever probe (22) with tip of sub-nanometric dimensions into close proximity with a sample (14) in order to allow

an interaction force to be established between probe (22) and sample (14);

(b) Applying a direct non-contact force (F_{direct}) to either the sample (14) or the probe (22) such that the probe (22) is encouraged to move towards the sample (14) or *vice versa*;

(c) Scanning either the probe (22) across the surface of the sample (14) or the sample (14) beneath the probe (22) whilst providing a relative motion between the probe (22) and surface such that an arrangement of scan lines covers the scan area;

(d) Measuring deflection of the cantilever probe (22); and

(e) Processing measurements taken at step (d) in order to extract information relating to the nanometric structure of the sample.

13. A scanning probe microscope (10) for writing information to a sample (14) by means of an interaction between the sample (14) and an AFM cantilever probe (22), the microscope comprising

driving means (16, 18, 20) arranged to provide relative scanning motion between the probe (22) and the sample surface and capable of bringing the sample (14) and probe (22) into close proximity; and

a probe writing mechanism arranged to vary intermittently, on a timescale shorter than one scan line, the strength of the interaction between the probe and the sample and so to change intermittently a property of the sample surface in the locality of the probe;

characterised in that, the microscope includes additional force (F_{direct}) generating means (24, 26) arranged such that, in operation, an

additional force (F_{direct}) is applied to either the sample (14) or the probe (22), the force (F_{direct}) being directed so as to attract the probe (22) towards the sample (14) or *vice versa*.

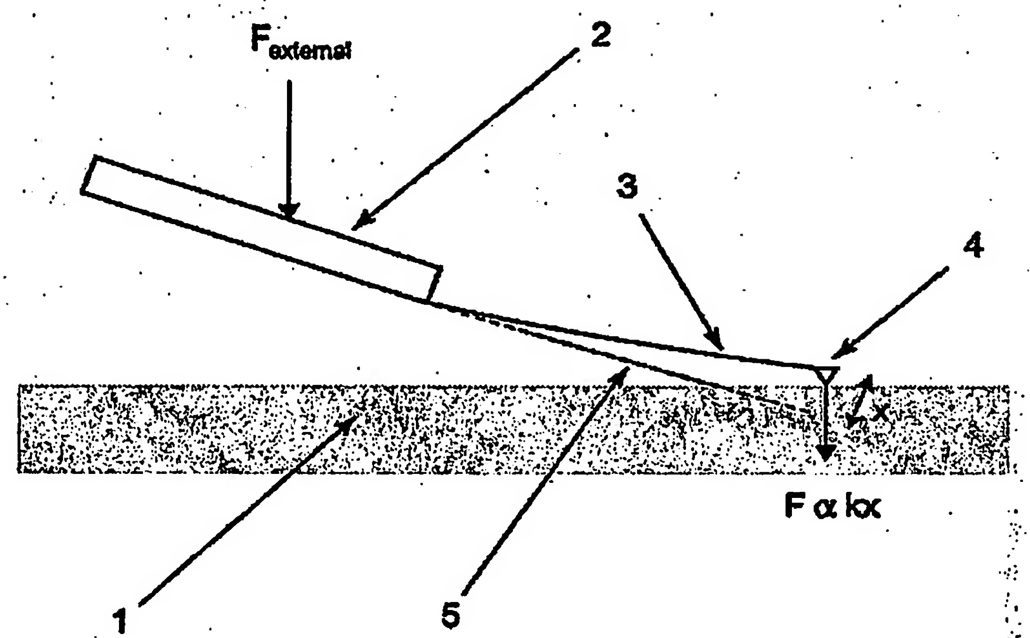


Fig 1

Prior Art

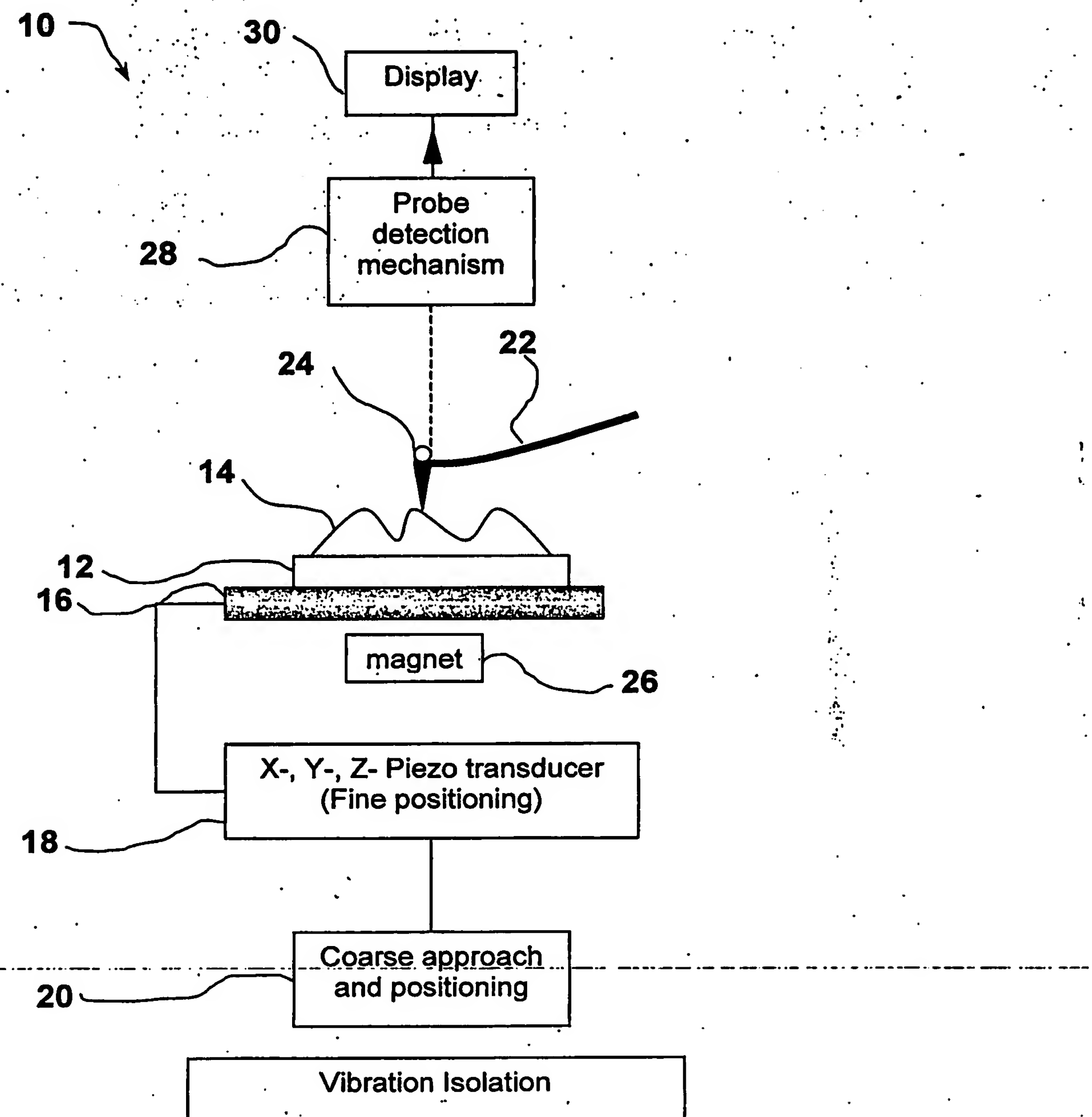
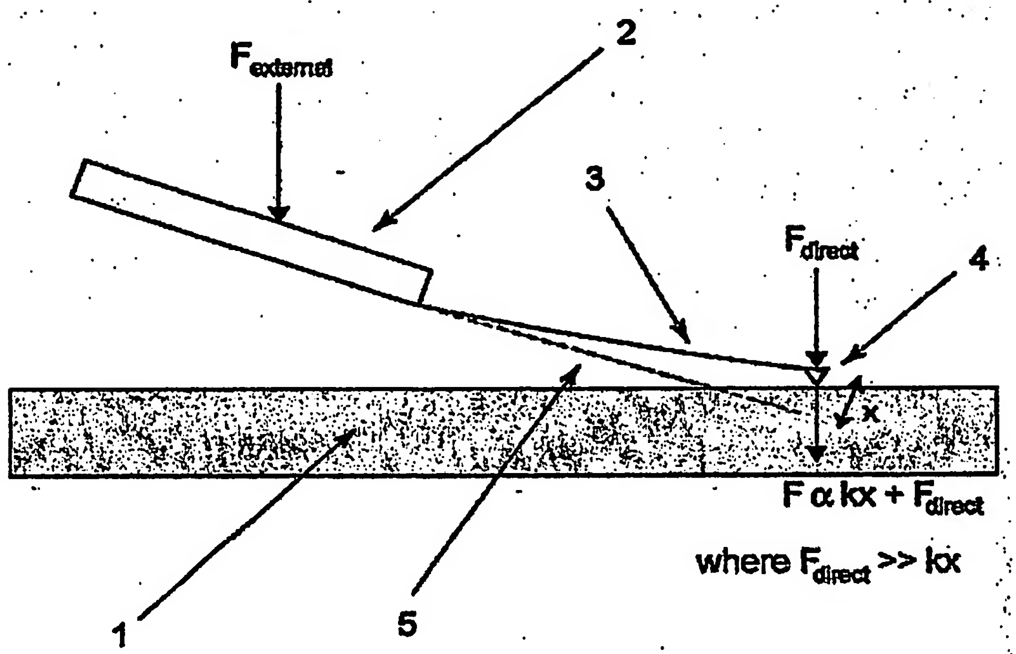

10
30
Display
Probe detection mechanism
28
24
22
14
12
16
magnet
26
X-, Y-, Z- Piezo transducer (Fine positioning)
18
Coarse approach and positioning
20
Vibration Isolation

Fig 2



$F_{external}$
2
3
F_{direct}
4
x
$F \alpha kx + F_{direct}$
where $F_{direct} >> kx$
1
5

Fig 3